

CENTERLESS GROUND THERMAL DESORPTION TUBE
AND METHOD WITHOUT FRIT

TO ALL WHOM IT MAY CONCERN:

BE IT KNOWN THAT ROBERT A HANDLY, citizen of the United States of America,
and resident of Harris County, City of Houston, State of Texas, has invented certain new
and useful improvements entitled as set forth above of which the following is a
specification.

Kenneth L. Nash, Esq.
Reg. No. 34,399
Law Offices of Kenneth L. Nash
P. O. Box 680106
Houston, TX 77268-0106
TEL: 281-583-1024
FAX: 281-397-6929
E-Mail: knash@houston.rr.com

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3 CENTERLESS GROUND THERMAL DESORPTION TUBE

4 AND METHOD WITHOUT FRIT

5

6 RELATED APPLICATIONS

7 U.S. Patent Application No. 10/368,204, filed February 17, 2003, and U.S. Patent
8 Application No. 10/717,810, filed November 20, 2003.

9

10 BACKGROUND OF THE INVENTION

11

12 (1) Field of the Invention

13 The present invention relates generally to systems and methods for area air
14 monitoring systems and, more specifically, to centerless ground DAMMS or thermal
15 desorption tubes.

16

17 (2) Description of the Prior Art

18 It is a common practice both in the military and industrial applications to
19 continuously monitor the atmosphere to detect, warn, monitor and/or verify the history
20 over long periods of time for the presence of toxic chemical agents and/or other chemical
21 compounds of interest that may exist in the area being monitored. Various types of

1 monitoring systems may utilize thermal desorption tubes, which are also referred to
2 herein as DAAMS (depot area air monitoring system) tubes.

3 Centerless ground desorption tubes are utilized in some types of fittings where it
4 is desirable that the centerless ground outer barrel fit exactly into the fitting to avoid
5 leakage between the fitting and the outer barrel. For these applications, it is desirable to
6 provide as perfectly round tube as possible which is produced by the process of centerless
7 grinding which is performed utilizing highly specialized equipment. The external
8 tolerance of centerless ground tubes is highly precise. These tolerances are usually less
9 than plus or minus 0.001 inches and may more typically range between plus and minus
10 0.0001 to .000050 or even 0.000001 inches, referred to hereinafter as a centerless
11 grinding tolerance.

12 In making centerless ground adsorbent tubes, it is desirable to provide a sturdy
13 mounting for sorbent or adsorbent material without damaging the close tolerances
14 produced by grinding. The industry accepted practice has been to utilize one or more
15 frits within the centerless ground adsorbent material. The frit is comprised of glass
16 grounds which typically are heated to thereby become affixed within the centerless
17 ground tube. In some cases, the fire polishing to mount the frit within the centerless
18 ground tube may cause slight changes to the tolerances at the position of the frit.
19 However, this technique has been accepted as an industry standard that has been used for
20 many years.

21 Fire polished fused glass spacers within the tubes to support the sorbent or
22 adsorbent material at a selected position, which are used for other types of applications,
23 have not been capable of being utilized with centerless ground adsorption tubes due the

1 tolerance changes caused by fire polishing/ fusing which are utilized to secure the glass
2 spacers within the outer barrel of the adsorption tube. Thus, the slight change in
3 tolerance due to fire polishing results in not being able to fit the tube exactly into the
4 fitting thereby causing unacceptable leakage between the fitting and the tube.

5 More generally, thermal desorption tubes may be utilized for the detection of any
6 chemical, biological, organism, or industrial warfare gas, matter, or agent. Thermal
7 desorption tubes typically utilize solid sorbents or adsorbents packed typically within a
8 glass tube. For some applications, stainless steel DAAMS tubes and glass-lined DAAMS
9 tubes have been utilized in the past. The air in the depot or environment is drawn through
10 the thermal desorption tube by a pump or blower over a predetermined time period
11 whereby the sorbent or adsorbent adsorbs the sorbate or chemical of interest in the air
12 which flows through the thermal desorption tubes. Thermal desorption tubes may be
13 used to detect even very low levels of the desired element in the air that may be at such
14 minute concentrations as to not be detectable by other means. The sample is then
15 thermally desorbed into a gas chromatograph for separation and detection typically
16 utilizing heating means, purge gases, filters, and the like. Use of the system is
17 advantageous in that it allows the trapping and concentration of a large volume sample in
18 a single sampling thermal desorption tube without the use of trapping solvents that would
19 otherwise dilute the sample. After desorption, the thermal desorption tubes are reusable
20 and ideally would generate virtually no waste.

21 Thermal desorption tubes have many applications and are often utilized for
22 historical monitoring whereby the history of the air may be measured to provide a record

1 of air history for a location going back for decades. In these cases, the thermal desorption
2 tubes are removed at regular intervals by personnel and placed into suitable desorption
3 equipment. On a daily basis, perhaps tens of thousands of thermal desorption tubes are
4 removed, purged, measurements recorded, and then replaced.

5 While as mentioned above, DAAMS tubes are known to have been made of
6 stainless steel and/or glass-lined stainless steel, this is not normally the case. High grade
7 stainless steel, e.g. 316 stainless steel, stainless steel is an expensive material. Stainless
8 steel and/or glass lined thermal desorption tubes are difficult to construct and expensive
9 to operate. Moreover, even very high grade stainless steel tends to be significantly less
10 chemically inert than glass, is electrically conductive, is subject to slight corrosion for
11 many chemicals, especially over long time periods, and subject to other problems that
12 may cause test contamination and/or problems.

13 Glass is much more commonly utilized for construction of the outer barrel of
14 thermal desorption tubes. The glass may comprise, for instance, borosilicate glass or
15 ground borosilicate glass. Adhesives are generally not used during construction because
16 of the potential problem of sample contamination caused thereby. Thermal desorption
17 tubes may utilize glass fusing to secure internal glass spacers that contain solid sorbent or
18 adsorbent and any necessary screens, or the like.

19 Other than stainless steel, less fragile materials such as plastics, polymers,
20 elastomerics, and the like, have much lower thermal degradation thresholds than glass or
21 stainless steel so as to break down or thermally degrade when heated to the thermal
22 desorption purging process temperatures that are typically and frequently utilized with

1 glass construction thermal desorption tubes. In this case, such materials would then give
2 off chemicals that could contaminate and thereby ruin or invalidate the sample.

3 The following U.S. Patents may be generally related to the above subject matter:

4 U.S. Patent No. 5,397,410, issued March 14, 1995, to R. A. Handly, discloses a
5 method for making machine readable bar code markings on glassware and is incorporated
6 herein by reference. A ceramic film first has bar code markings placed thereon. The
7 ceramic film with the bar code markings is placed on the outer surface of the glassware.
8 The ceramic film and glassware are heated or fired for a predetermined time period at a
9 temperature over 1100 degrees Fahrenheit adequate to effect fusing of the film onto the
10 glassware while providing a fused layer therebetween.

11 U.S. Publication No 2003/0067393 A1, published April 10, 2003, to Albro et al.,
12 discloses a system for the collection of near real time confirmation samples is provided to
13 quickly eliminate false positive alarms by confirming the presence or absence of a
14 chemical agent when a monitor operating in near real time to detect the presence of that
15 chemical agent generates an alarm. The confirmation sampling system is synchronized
16 with the near real time monitor and the confirmation sampler and monitor draw common
17 samples of the atmosphere of concern. In the event that the monitor generates an alarm,
18 the confirmation sampler preserves the sample taken contemporaneously with the alarm
19 event for separate analysis, and also takes and preserves one or more follow-on samples.

20 U.S. Patent No. 5,922,443, issued July 13, 1999, to Larsen et al., discloses a
21 polymeric article and method for making a polymeric article, such as a catheter, are
22 disclosed wherein the article is contacted with a swelling agent, such as a solvent, and a

1 plasticizer to make the polymeric material softer and/or more pliable or flexible. The
2 contacting may be carried out sequentially or simultaneously, using a solution containing
3 a plasticizer and a solvent.

4 U.S. Patent No. 4,885,500, issued December 5, 1989, to Hansen et al., discloses A
5 quartz quadrupole comprises a quartz substrate, conductive strips and low-conductivity
6 strips. The substrate includes hyperbolic inner surfaces which provide the geometry for
7 the conformed conductive strips to produce an appropriate electric field for mass filter
8 operation. The use of quartz as a substrate material provides the thermal and electrical
9 characteristics required by high performance mass filtering operations, including
10 scanning mode operation to 800 amu and above. During such operation, potential field
11 distortions by accumulated charge in cusp sections of the substrate are minimized by the
12 low-conductivity strips, which are arranged to overlap longitudinal edges of the
13 conductive strips. Formation of the quartz substrate is made possible by high precision
14 machining, grinding and polishing of a refractory metal mandrel. The actual step of
15 forming the substrate is simplified by the low thermal coefficient of expansion of the
16 quartz. The conductive strips are applied by firing metal-glass frit tape. The low-
17 conductivity strips are applied by firing a metal-oxide slurry including a bonding agent.

18 U.S. Patent No. 4,021,219, issued May 3, 1977, to Stockdale et al., discloses A
19 stacking method is employed to assemble a structure which includes a plurality of
20 substantially identical, uniformly spaced parallel glass plates. The method includes the
21 use of a fixture having a slotted surface in which rigid cylindrical spacers can be slidably
22 disposed. Each glass plate is placed with its edge surface on the slotted surface and
23 spaced from the adjacent plates by at least two of the cylindrical spacers, each of the

1 spacers being disposed in a separate slot. Reference planes which are perpendicular to the
2 surface of the fixture are provided so as to maintain the plates in the desired spatial
3 relation. Then, a glass base is bonded onto the opposing edge surface of each of the
4 spaced glass plates. The glass plates and glass base are preferably chosen to be of the
5 same material. The spacers are chosen to be of a material having an expansion coefficient
6 slightly greater than that of the glass plates and glass base.

7 The above patents do not provide a centerless thermal desorption tube without a
8 frit. The inventor believes that a disadvantage of the use of the centerless ground tubes
9 with a frit is that the ground glass frit and/or heating utilized to mount the frit therein
10 produces a pressure drop which is not consistent from tube to tube. Therefore flow
11 through the tube may be unpredictable or variable from tube to tube. This may cause
12 inaccuracies in measurement. Accordingly, a centerless ground tube is provided as
13 taught herein which provides a support for the sorbent or adsorbent material without the
14 use of one or more frits. Those skilled in the art will appreciate the present invention that
15 addresses the above and other problems.

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1 between the inner spacer and the outer barrel to secure the inner spacer with respect to the
2 outer barrel. A sorbent material is positioned within the outer barrel. In one preferred
3 embodiment, either or both of the outer barrel or the inner spacer(s) comprise a
4 fluoropolymer material, a non-limiting example being PTFE.

5 The thermal desorption tube may further comprise glass wool plugs positioned
6 between at least two inner spacers with the sorbent material being positioned between the
7 glass wool plugs. Additionally, the thermal desorption may further comprise at least two
8 screens positioned outside the glass wool plugs but inside the spacers.

9 The outer barrel and the inner spacer(s) may be comprised of the same material or
10 of different materials. In a preferred embodiment, at least one of the outer barrel or the
11 spacer(s) may be comprised of a resilient material.

12 In another embodiment, the thermal desorption tube, may comprise one or more
13 elements such as, for example, an outer barrel comprised of a non-glass and non-steel
14 material a sorbent material positioned within the outer barrel. The material of the outer
15 barrel may preferably be selected such that a maximum temperature required for a
16 thermal desorption process of the sorbent material to which the outer barrel is to be
17 heated during the thermal desorption process is less than a thermal degradation threshold
18 of the material.

19 In yet another embodiment, the inner spacer(s) and the outer barrel may be sized
20 so as to be held in position by friction after an insertion of spacer(s) within the outer
21 barrel. At least prior to construction an inner diameter of the outer barrel may be slightly
22 less than or equal to an outer diameter of the inner spacer(s) such that sufficient friction is

- 1 produced by an interference fit therebetween to secure the inner spacer(s) within the outer
- 2 barrel. Alternatively, the inner diameter may be slightly greater than the outer diameter
- 3 of the inner spacer(s) and sufficiently close in tolerance, length, and so forth, to produce
- 4 sufficient friction to hold the assembly together.

1 BRIEF DESCRIPTION OF THE DRAWING

2 A more complete understanding of the invention and many of the attendant
3 advantages thereto will be readily appreciated as the same becomes better understood by
4 reference to the following detailed description when considered in conjunction with the
5 accompanying drawing wherein corresponding reference characters indicate
6 corresponding parts throughout the drawing and wherein:

7
8 FIG. 1 is an enlarged elevational view, in cross-section, of a centerless ground
9 thermal desorption tube in accord with one embodiment of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring now to the drawings and, more particularly, to FIG. 1 there is shown thermal desorption tube 10 in accord with the present invention.

As a general overview, thermal desorption tube 10 may comprise, in one possible embodiment, centerless ground outer barrel 12, one or more inner spacers such as inner spacers 14 and 16, one or more screens such as screens 18 and 20, inner glass wool plugs 24 and 26, outer glass wool plugs 28 and 30, and adsorbent or sorbent material 22.

Various modifications of this basic construction may be utilized. For instance, multiple sorbent beds may be utilized which may be separated, if desired, by additional glass wool plugs and/or screens. Depending on the applications and types of adsorbent or sorbent material 22, stainless steel screens 18 and 20 may or may not be utilized and/or silanized glass wool plugs may or may not be utilized, as desired, for construction of thermal desorption tube 10.

In a preferred embodiment, centerless ground outer barrel 12 is comprised of borosilicate glass that has been centerless ground. Other centerless ground materials for outer barrel 12 include, but are not limited to: simax glass, Pyrex glass, quartz tubing fused quartz, synthetic silica. As used herein, these materials are referred to generally as glass. Centerless ground outer barrel 12 also could have manufacturing variations such as being extruded, dipped, coated, impressed, reinforced, wrapped, and/or sanitized. Centerless ground outer barrel 12 in accord with the present invention may be high purity labeled, have various wall thicknesses, may be tapered, colored, translucent, have raw ends, or may be fire polished at selected positions. Generally, use of other materials for

1 outer barrel 12 are not desirable unless they are such that they can have a tolerance that is
2 acceptable to provide a suitable seal when inserted into the fitting utilized by the
3 customer.

4 Inner spacers 14 and 16 are preferably comprised of the same material as
5 centerless ground outer barrel 12. In some cases, only one inner spacer may be used.
6 If desired, the material of spacers 14 and/or 16 might be different from that of barrel 12
7 and might conceivably comprise non-glass materials as may be discussed in more detail
8 in one or more of my previous application such as, for instance, related applications U.S.
9 Patent Application No. 10/368,204, filed February 17, 2003, and U.S. Patent Application
10 No. 10/717,810, filed November 20, 2003, all or which are incorporated herein by
11 reference. Generally, while it is generally desirable to utilize the same materials, non-
12 glass materials such as PTFE or the many non-glass materials listed in the above
13 applications might possibly provide a fluid (gas) flow sealed friction fit, depending on the
14 application, to avoid the need for fire polishing and/or any type of adhesives. Moreover,
15 different material spacers may be used on either end of thermal desorption tube 10
16 depending on the possible usage therefore, although in a presently preferred embodiment
17 if two spacers such as 14 and 16 are utilized, and/or other spacers, then the spacers would
18 be comprised of the same material.

19 Generally, it is not necessary that inner spacers 14 and 16 are centerless ground,
20 but one or more of the inner spacers could be centerless ground, if desired. For instance,
21 centerless ground inner spacers might possibly be utilized to provide a fluid (gas) flow
22 sealed friction fit with a glass outer barrel 12.

1 In one presently preferred embodiment, at least one inner spacer, such as inner
2 spacer 14, is fire polished at end 32 before outer barrel 12 has been centerless ground.
3 The fire polishing of end 32 firmly secures inner spacer 14 within outer barrel 12 and
4 prevents any leakage between inner spacer 14 and outer barrel 12. After inner spacer 14
5 is fire polished so as to be secured within outer barrel 12, then outer barrel 12 is
6 centerless ground, including at end 32, to provide a virtually perfectly round centerless
7 ground outer barrel without the need for a frit to hold sorbent material 22 and/or the
8 screens and/or glass wool in place within outer barrel 12. The perfectly round thermal
9 desorption tube 10 will then provide a perfect mating surface to the fitting. Generally,
10 only one inner spacer will be fire polished to secure it in place prior to having outer barrel
11 12 centerless ground. This is because one end of thermal desorption tube 10 may extend
12 outwardly from the fitting and therefore it is not so important that the end not extending
13 out of the fitting be perfectly round, such as due to fire polishing. Thus, the second inner
14 spacer may be fire polished after outer barrel 12 is centerless ground. In some cases, a
15 second inner spacer may not be utilized at all. As well, a different material spacer could
16 be utilized with a friction fit as discussed above and in the related applications discussed
17 hereinbefore.

18 However, it is also possible, after fire polishing the first inner spacer in position,
19 and before the centerless grinding process, to completely construct the desired thermal
20 desorption tube including any sorption material sections 22, screens, glass wool, and the
21 like, and then fire polishing the second inner spacer. In this case, both inner spacers 14
22 and 16 may be fire polished prior to centerless grinding of outer barrel 12, at both ends 32

1 and 34. However, this method has the disadvantage that the sorbent material will be
2 heated during the centerless grinding process. It is usually desirable that the sorbent
3 material not be heated prior to sale to a customer. Nonetheless, the heating of the sorbent
4 during the centerless grinding process may not be of importance to some customers.

5 While desorption tube 10 is shown as being straight, please note that desorption
6 tube 10 may include bent sections or u-shaped sections if desired and depending on the
7 types of fittings and/or other equipment with which desorption will be utilized.

8 As yet another option, if desired, the at least one of spacers 14 and/or 16 could be
9 melted for securing spacers 14 and/or 16 in position within a centerless ground outer
10 barrel 12 and/or special adhesives suitable for particular applications might be utilized
11 and/or other mounting means may be utilized. If at least one of spacers 14 and/or 16
12 were comprised of non-glass material for friction fitting, such as PTFE, the wall
13 thickness and length of spacers 14 and 16 will be factors that affect the friction produced
14 by construction thereof in this manner. If PTFE or other non-glass materials are used for
15 spacers 14 and/or 16, then spacers 14 and/or 16 might also be slightly tapered so as to be
16 insertable to provide a somewhat compressed engagement to ensure a seal. The smallest
17 diameter of the taper might then be less than an inner diameter of the outer barrel 12 but
18 may increase to the slightly greater than the inner diameter of spacers 14 and 16 to permit
19 insertion therein. The same tapering process might be utilized for friction sealing of glass
20 spacers within outer barrel 12. Glass spacers are less compressible but may be designed
21 to still tightly and sealingly fit together.

1 In another embodiment, depending on the materials utilized for barrel 12 and
2 spacers 14 and/or 16, an interference fit may be utilized wherein the outer diameter of
3 spacers 14 and 16 may be slightly greater than the inner diameter of barrel 12.

4 In a presently preferred embodiment, thermal desorption tube 10 may be easily
5 constructed. For instance, spacer 16 may be inserted into outer barrel 12 and fire
6 polished to secure spacer 16 in position. Generally, it is desirable that the end of spacer
7 16 be directly adjacent the end of outer barrel 12 as shown in FIG. 1. Conceivably, this
8 arrangement could be varied.

9 Then outer barrel 12 with fire polished inner spacer 16 is centerless ground so as
10 to be round to a great degree of precision. Screen 18 may be inserted, if desired. Then, if
11 desired, glass wool plugs 28 and 30 may be inserted into outer barrel 12. Adsorbent or
12 sorbent material 22 may be added. Glass wool plugs 26 and 28, and screen 20 may then
13 be added. Then spacer 14 may be inserted and fire polished to secure it in position.
14 Because spacers 14 and 16 are fixedly mounted with respect to barrel 12, spacers 14 and
15 16 easily hold the internal components in position. The fit is quite tight and sturdy. If
16 end 32 is fire polished and then centerless ground, any suitable type of marking may be
17 utilized to provide the customer with information as to which end is to be inserted into
18 the fitting.

19 Barrel 12 is preferably of high purity and hardness. Sorbent or adsorbent material
20 22 may comprise but is not limited to: Tenax gr, Tenax ta, Chromasorb p, Chromosorb
21 100-108, Carbowax, Porapak, Hayesep A-Z, Carbosieve, Carboxen, Carbotrap, Silica gel,
22 Charcoal, Acid washed glass beads, Carbopack, Molecular sieve, Anasorb GCB1,

1 Anasorb GCB2, Carbosieve S-111, Anasorb CMS. If desired, and depending on the type
2 of thereof, sorbent or adsorbent material 22 may be positioned within outer barrel 22
3 without use of screens or glass plugs and held in place by friction therewith. Other means
4 to mount sorbent or adsorbent material 22 may be utilized. Sorbent or adsorbent material
5 22 may comprise fibrous polymer material. Sorbent or adsorbent material 22 may be
6 formed in various shapes and constructions and may or may not comprise layers,
7 substrates, woven portions, non-woven portions, tubular portions, powders, and the like.

8 One, two, or more screens such as screens 18 and/or 20, if utilized at all, may
9 comprise materials such as the following but are not limited to: stainless steel, aluminum,
10 brass, niobium, copper, gold, hastelloy, inconel, kanthanl, molybdenum, monel, mp35n,
11 nichrome, nickel, bronze, platinum, silver, tantalum, titanium, tungsten, any diagonal, flat
12 wire, hex or interchimp mesh, any market grade, raw edge, cut edge, or any selvage, any
13 cloth thickness from .0005 to .9999, any twilled filter cloth weave, plain filter cloth
14 weave, twilled weave, plain weave, unfc, micron weave, or other types of weaves.

15 One or more glass plugs such as plugs 24, 26, 28 and/or 30, if utilized at all, may
16 comprise materials such as the following, but are not limited to: any borosilicated treated
17 glass wool including Pyrex, simax, quartz, soda lime, kimax, synthetic silica, or Duran.
18 The wool plugs product name may be Pyrex. Synonyms for this type of product include:
19 angel hair, fiberglass, chemical name: borosilicate glass wool, silylated. The glass wool
20 may be treated with a silylation reagent to react with the silanol groups to make it more
21 suitable for its intended application in chromatography.

22 As shown in previous applications, a bar code (not shown) or other label which

1 may be utilized to automate the recording process for recording the results, times, and
2 other information related to use of thermal desorption tube 10. The bar code, if used,
3 may also provide an indicator of which end of centerless ground outer barrel 12 to insert
4 into the fitting.

5 Common sizes of barrel 12 may comprise but are not limited to an outside
6 diameter of barrel 12 ranging from about 1/8 inches to about one inch. The minimum
7 inside diameter of thermal desorption tube 10 may typically but not necessarily range
8 from about 0.0390 inches to about one inch. The length of the adsorbent bed or beds may
9 typically range from about 0.0390 inches to about seven inches. The length of barrel 12
10 may typically but not necessarily range from 0.0390 inches to about twenty-four inches.
11 Spacers 14 and/or 16 may typically but not necessarily range in length from about 0.390
12 inches to about twelve inches.

13 Therefore, it will be understood that many additional changes in the details,
14 materials, steps and arrangement of parts, which have been herein described and
15 illustrated in order to explain the nature of the invention, may be made by those skilled in
16 the art within the principle and scope of the invention as expressed in the appended
17 claims.